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Control of Sulfur Emissions from Oil Shale Retorting Using Spend Shale Absorption

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K. D. Van Zanten and G. R. Chiaramonte (J and J Associates); and E. R. Bates (EPA/AEERL)

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18200 W. Highway 72
Golden, Colorado 80401

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16. ABSTRACT The paper gives results of a detailed engineering evaluation of the potential for using an absorption on spent shale process (ASSP) for controlling sulfur emissions from oil shale plants. The evaluation analyzes the potential effectiveness and cost of absorbing SO2 on combusted spent shale in either a fluidized-bed combustor (FBC) or a fluidized transport system (lift pipe) for three retorting processes: Lurgi (lift pipe), Union C (FBC), and Modified In-Situ (FBC). Conceptual designs were completed for the three retorting processes, and the costs of removing SO2 by the ASSP approach were compared to the costs of removing either H2S (including other sulfur species such as mercaptans and thiophenes) or SO2 by the leading conventional sulfur removal processes. A number of significant process variables were identified and evaluated in the study. Study results indicate that ASSP is technically and economically viable compared to conventional technologies for most retorting processes. However, some technical uncertainties will remain until further testing is conducted. Environmental acceptability depends not only on the effectiveness of ASSP for controlling sulfur emissions but also on holding NOx emissions to a low level and on not volatizing significant quantities of trace elements such as mercury and cadmium.

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Oil Shale	Nitrogen Oxides	Stationary Sources	08G
Retort Furnaces		Absorption on Spent	13I, 13A
Absorption		Shale Process (ASSP)	14G
Desulfurization		Spent Shale	07A, 07D
Sulfur Oxides			07B

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Control of Sulfur Emissions From Oil Shale Retorting Using Spend Shale Absorption

Kenneth D. Van Zanten and Gerald R. Chiaramonte

J & A Associates, Golden Colorado

and

Edward R. Bates

United States Environmental Protection Agency, Cincinnati, Ohio

A detailed engineering evaluation of the potential for using an absorption on spent shale process (ASSP) for controlling sulfur emissions from oil shale plants was completed. The evaluation analyzes the potential effectiveness and cost of absorbing SO_2 on combusted spent shale in either a fluidized bed combustor (FBC) or a fluidized transport system (lift pipe) for three retorting processes: Lurgi (lift pipe), Union C (FBC), Modified In Situ (FBC).

Conceptual designs were completed for the three retorting processes and the costs of removing SO_2 by the ASSP approach were compared to the costs of removing either H_2S (including other sulfur species such as mercaptans and thiophenes) or SO_2 by the leading conventional sulfur removal processes. A number of significant process variables were identified and evaluated in the study.

The study was performed under contract with the U.S. Environmental Protection Agency.

INTRODUCTION

Control of sulfur emissions is a key environmental concern in the retorting of oil shale. Oil shale may contain 2% or more sulfur, while a typical shale in the Green River Formation of Colorado contains about 0.7% sulfur. Although this sulfur concentration is not large compared to some high sulfur coals (4 to 8% sulfur), large-scale production of shale oil will require processing millions of tonnes of oil shale; thus, sulfur emissions would be very great if controls were not applied. About 16 to 30% of the sulfur in oil shale is liberated to the vapors (oil and gas) produced during retorting, and the remainder stays with the spent shale. Retort gases generally contain hydrogen sulfide (H_2S) as the major sulfur compound and lesser amounts of other organic sulfur compounds such as sulfur dioxide (SO_2), carbonyl sulfide (COS), carbon disulfide (CS_2), and mercaptans (CH_3SH and C_2H_5SH).

To date, the federal government has not promulgated NSPS (New Source Performance Standards) for the oil shale industry. However, the U.S. Environmental Protection Agency does regulate SO_2 (and some other emissions) through PSD (Prevention of Significant Deterioration) permitting requirements of the Clean Air Act. This legislation requires use of BACT (Best Available Control Technology—costs considered) to minimize the addition of specified pollutants in order to preserve air quality. PSD permits are issued on a facility by facility basis after careful analysis of each application. The federal PSD requirement that generally is most difficult for developers to meet is the 24 hour Class I air quality standard limiting SO_2 to 5 $\mu g/m^3$. In addition to federal requirements, states also control the emission of SO_2 from oil shale facilities. For example, the State of Colorado limits SO_2 emissions to less than 0.86 kg SO_2/m^3 (0.3 lb/bbl) for shale oil produced.

Control of sulfur emissions has been estimated to constitute a major portion of the environmental control cost for oil shale facilities. For example, the Denver Research Institute has estimated costs in the range of \$1 - 3/bbl of shale oil produced [*1, 2*]. These substantial sulfur control costs have encouraged developers to seek less costly but equally or more effective methods for limiting sulfur emissions. Recently, a strong industry trend has been to look toward the potential for combusting carbon on carbonaceous retorted shale to recover its energy value (a plus in terms of economics and resource conservation), while exploring the possibility of absorbing the sulfur gases produced during retorting onto the calcined carbonate material present after combusion of retorted western oil shale. In order to obtain more information to support its PSD permitting decisions on sulfur control and in order to investigate whether emission of other pollutants such as nitrogen oxides (NO_x) and trace elements might be significantly increased, EPA awarded a contract to J & A Associates to investigate the environmental advantages/disadvantages of absorbing SO_2 onto combusted retorted oil shale.

CONVENTIONAL CONTROL TECHNOLOGIES

Two approaches have generally been used to control sulfur emissions from retort gases—removal of the sulfur compounds from the gas before it is combusted (gas sweetening) or combustion of the gas followed by removal of the SO_2 from the flue gases (flue gas desulfurization).

Gas sweetening processes such as amine absorption and Stretford generally are very effective in removing H_2S. However, organic sulfur compounds, principally COS, are not significantly removed or are only partly removed by these processes. While H_2S levels are reduced to about 10 ppmv in the sweet gas, overall sulfur removal efficiencies are lower (95-98%) because the organic sulfur compounds are not completely removed. Flue gas desulfurization (FGD) processes, which use an aqueous slurry of lime to absorb SO_2 from the flue gas, generally are not as highly effective in removing SO_2. Typical SO_2 removal efficiencies range from 90 to 95%. In addition, FGD processes must handle larger volumes of gas than gas sweetening processes.

To meet Colorado's air quality regulations of 0.86 kg SO_2/m^3 of shale oil produced (0.3 lb/bbl), about 95-96% of the total sulfur in the gas would have to be removed. This makes use of FGD processes marginal and tends to focus more interest on gas sweetening processes. However, gas sweetening plants are generally expensive to build and operate, and the sulfur product recovered may not be saleable because of product impurities or the remote location of the oil shale plants from traditional sulfur markets.

The practicality of conventional control technologies applied to oil shale plants depends to a great extent on the type of retorting process employed, i.e., direct heated vs. indirect heated. Direct heated retorts are those in which direct combustion with air occurs inside the retort to supply the heat of retorting. Examples of such processes are Modified In-Situ (MIS), Paraho Direct Heated, and Union A. Indirect heated retorts provide heat using heated gases or solids and no combustion occurs in the retort vessel. Examples are Union B and C, Tosco II, Lurgi, and Chevron STB.

In direct heated retorts, sulfur is liberated mostly as H_2S, but the off-gases are relatively dilute in hydrocarbons because large quantities of N_2, O_2, CO_2, and H_2O are liberated from combustion. H_2S removal from such gas streams is quite expensive using conventional gas sweetening technologies and in some cases is economically prohibitive. Non-selective process (i.e., processes which co-sorb both H_2S and CO_2) are unattractive because of the very great cost associated with removing the large quantities of CO_2. Selective processes (selective for H_2S), such as Stretford, are more economically attractive, but there remain numerous technical concerns about Stretford-type processes with regard to solvent degradation, quality of product sulfur, spent solvent disposal, etc.

From a sulfur emissions viewpoint, indirect heated retorts are less problematic with respect to gas treatment, but there are a number of problem areas. Indirect heated retorts produce gases with relatively little CO_2 (although higher than natural or refinery gases), and don't have O_2 or N_2 as diluents. Proposed treating plants for such gases use conventional processes such as amine sweetening or other processes typically applied to handling natural or refinery gases. Typically, regenerated H_2S streams end up in a Claus sulfur plant with tail gas treatment. Design of a Claus plant, applied to oil shale gas treating, is complicated by quantities of both NH_3 and CO_2, which are higher than in natural or refinery gases. Virtually none of the conventional gas sweetening processes do much to remove sulfur species other than H_2S, e.g., COS, CS_2, and mercaptans. For the most part, these stay in the gas which, when burned, end up as SO_2 in the stack. All of these conventional treating plants are more expensive to build and operate than comparable systems in refineries and gas plants because of the higher levels of CO_2, and to a lesser extent, because of higher levels of NH_3 and unsaturated hydrocarbons.

THE ASSP CONCEPT

Retorted shale contains from 4 to 10 wt% organic carbon, and research efforts have been directed to recovering this energy by combusting the shale, usually in a fluid bed or some other fluidized transport device. Initially, there was some concern that excessive sulfur emissions might result because the retorted shale contains approximately 0.7 wt% sulfur in the form of organic sulfur, pyrite (FeS_2), and/or pyrrhotite (FeS). However, it was soon discovered that sulfur emissions were very low when combusting carbonate-containing retorted shale. Approximately half the mineral content of Green River oil shale consists of dolomite [$CaMg(CO_3)_2$] and calcite ($CaCO_3$). These two minerals are the essential components of calcareous rocks used in controlling SO_2 in coal combustion. These carbonates in oil shale are fine-grained, with grain sizes ranging from 1 to 10 μm which is desirable since fine-grained calcareous rocks are generally better SO_2 sorbents than the coarse grained rocks.

A number of laboratory and small pilot scale experiments have been conducted to test the ability of carbonate-containing shales to control sulfur emissions. For example, McCarthy [3] conducted experiments in a 0.1 m (4 in.) diameter fluidized bed burning raw shale fines and sulfur containing gases. He found that at 1083-1093 K (810-820°C), a 0.2 v% H_2S gas could be burned, emitting less than 1 ppmv sulfur as SO_2.

This ability of combusted carbonate-containing spent shale to absorb SO_2 gives rise to a novel concept for controlling sulfur emissions in oil shale plants. This concept will be referred to as ASSP which stands for Absorption on Spent Shale Process. The ASSP concept has several potential advantages over conventional sulfur removal technologies:

- The sorbent is cheap and inherently abundant in oil shale plants.
- The process requires combustion of the spent shale which is already incorporated into several of the retorting technologies or which would be a useful add-on to recover residual carbon values.
- Since non-H_2S compounds are converted to SO_2 by combustion, ASSP could represent a more efficient removal relative to gas sweetening processes which only remove H_2S.
- The process does not present any incremental disposal requirements.

This paper discusses preliminary results of a study commissioned by the U.S. Environmental Protection Agency to assess the viability of the ASSP concept relative to conventional technologies as applied to control of sulfur emissions from oil shale plants. The program, which is under contract to J & A Associates, is being done in two phases. Phase I provides an engineering assessment and cost for application of ASSP to selected leading retorting processes. Phase II conducts experimental work in an integrated oil shale pilot plant to define operability and proof of principle. The Phase I work has been completed and is summarized here. The Phase II work is scheduled for the 4th quarter 1985 and will be reported later.

KEY PROCESS VARIABLES

The process variables which affect the ability of combusted shale to absorb SO_2 are:

Temperature
Calcium to sulfur ratio
Mineral content of the shale
Extent of carbonate decomposition
Use of raw shale vs. spent shale as feed
Solids residence time

Particle size
Gas residence time
Gas composition
Oxygen concentration
Water concentration
Solids/gas mixing (mass transfer)

Temperature, Ca/S ratio, carbonate decomposition, solids residence time, particle size, and gas residence time are probably the most important variables. Of these, temperature and Ca/S ratio are believed to be the key independent variables

PROCESS DESCRIPTION

The ASSP concept uses a fluidized transport system to combust either raw or retorted shale, thereby providing the vehicle for converting sulfur compounds to SO_2 and absorbing the SO_2 in the shale matrix. The concept envisions either a conventional dense-phase fluidized bed or a dilute-phase contactor (lift pipe). Key elements of the process are shown in Fig. 1.

Solids feed is either crushed raw shale or retorted shale with a top size of 0.0064 m (1/4-in.) or a combination of raw and retorted shale. Air is introduced at the bottom of the contactor along with process and/or waste gases to be treated. The contactor is provided with coils for removing heat. Heat removed is used either for heating process gas streams or for raising steam. Cyclones, flue gas coolers, scrubbers, and/or bag houses are provided for recovering heat and removing dust from the combustor flue gas. NOx emissions are controlled by staged combustion; i.e., an oxygen-lean first stage followed by an oxygen-rich second stage. Spent ash is discharged and cooled for heat recovery and moisturized prior to disposal.

CONCEPTUAL DESIGN BASIS

The engineering assessment evaluated the ASSP concept as applied to three types of retorting processes:

Direct heated
Indirect heated
Indirect heated with combustor integrated into the process

For study purposes, specific retorting technologies and sites were selected as representative of these three retort types:

Retort Type	Process	Site
Direct heated	MIS with Unishale C	Cathedral Bluffs (Tract C-b)
Indirect heated	Unishale B	Union
Integral combustor	Lurgi	Rio Blanco (Tract C-a)
	Unishale C*	Union

*The Unishale C Process is Unishale B with the addition of a fluid bed combustor.

Simplified block flow diagrams for each study case are given in Figures 2, 3 and 4.

The study assumed that an amine process is used to remove acid gases (H_2S and CO_2) from indirect heated retort gases. The regenerator acid gases are then processed in either a fluidized bed combustor (Unishale C process) or a

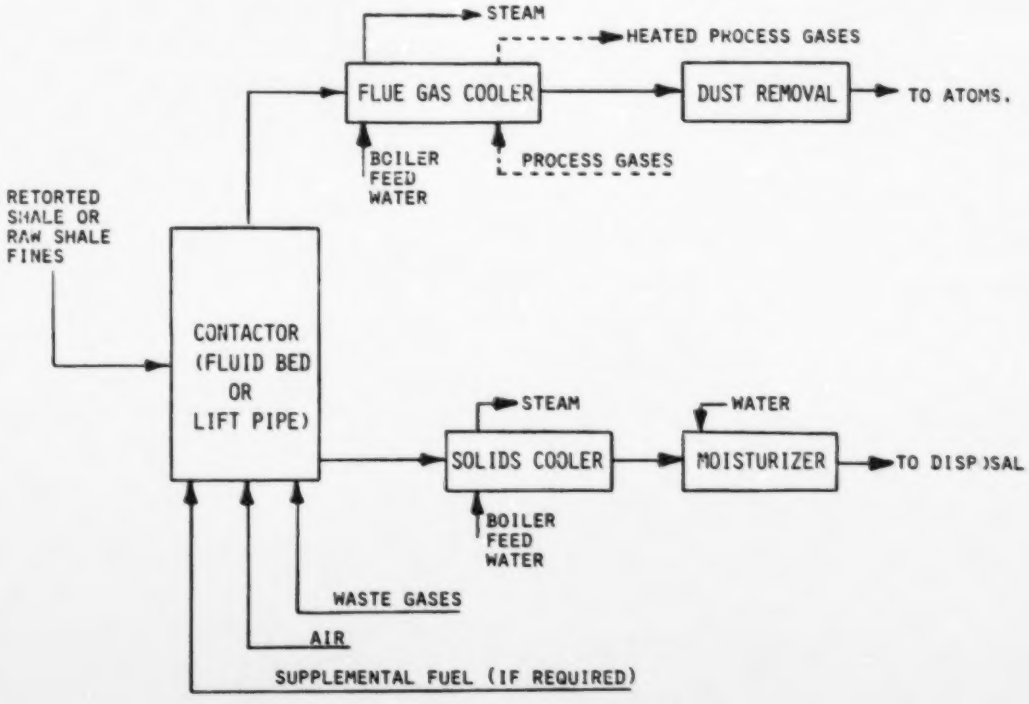

Figure 1. ASSP process flow diagram.

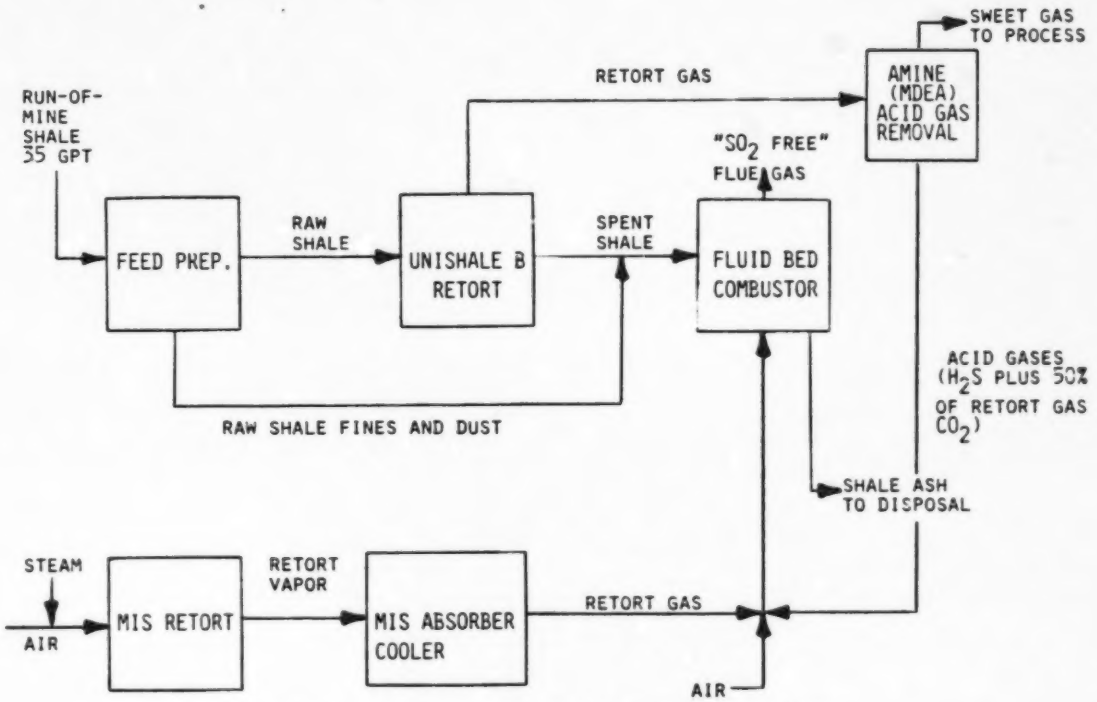

Figure 2. Process flow diagram for ASSP evaluation. Direct heated (Modified In-Situ with Unishale C).

lift pipe combustor (Lurgi process) where spent shale and/or raw shale fines are burned to supply some of the retorting heat requirements. Modified in-situ retort gases are processed directly in a fluidized bed combustor without any pretreatment. For each type of retort studied, a conceptual process design was developed to evaluate the potential effectiveness and cost of applying the ASSP process on a commercial scale. Conventional sulfur removal

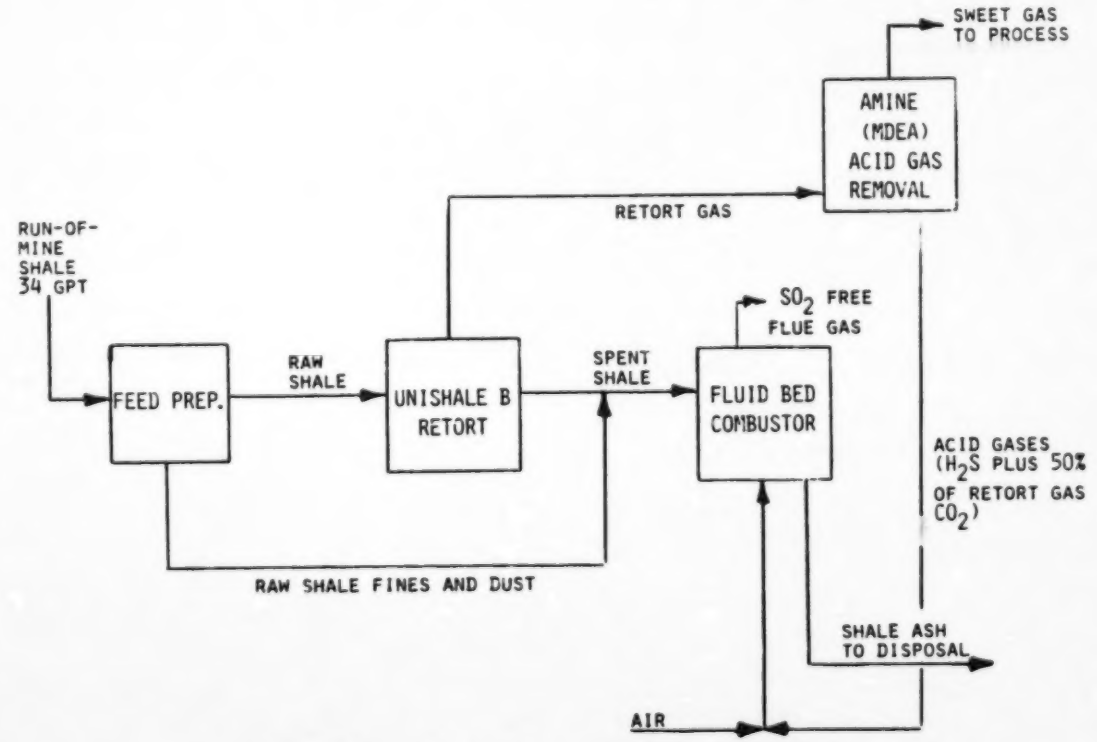

Figure 3. Process flow diagram for ASSP evaluation. Indirect heated (Unishale B).

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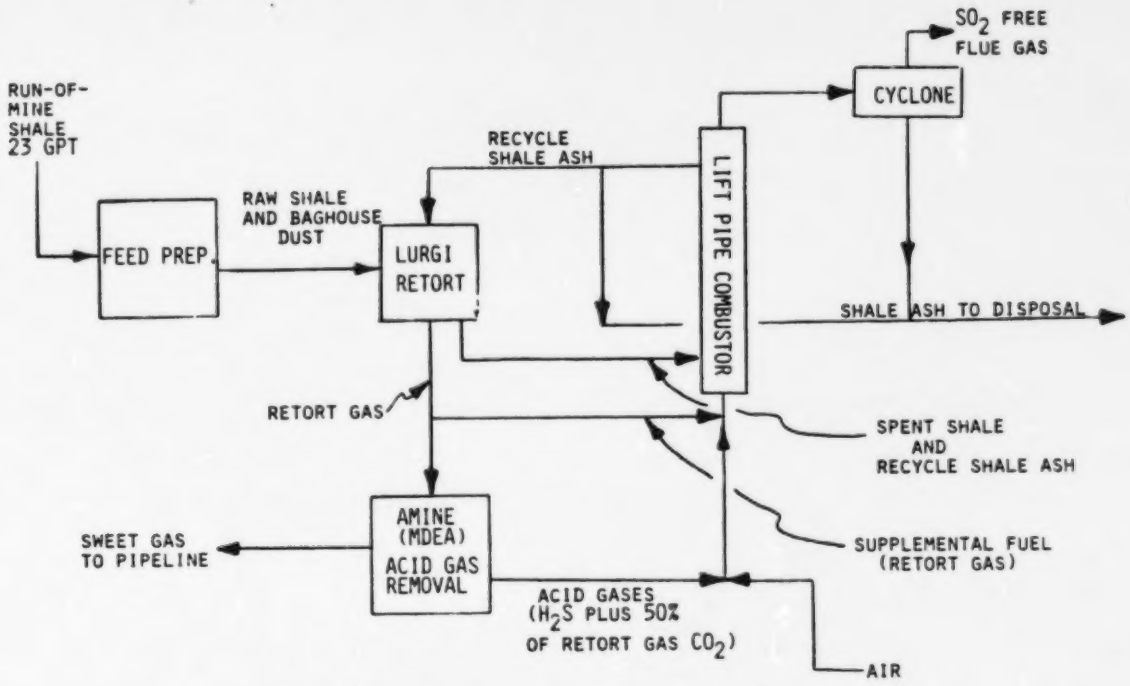

Figure 4. Process flow diagram for ASSP evaluation. Integral part of process (Lurgi).

processes, which are either being planned or are currently in use, were also evaluated. The capital and operating costs of the ASSP process were then compared to these conventional sulfur removal processes. Conventional processes evaluated were:

Retort Type	Retort Process	Conventional Treating Process
Direct heated	MIS + Unishale C	Case A: Unisulf + Flue Gas Desulfurization on MIS gases Case B: Unisulf + Stretford on MIS gases
Indirect heated	Unishale B	Unisulf*
Integral combustor	Lurgi Unishale C	Diethanolamine (DEA) + Stretford Unisulf

*Unisulf is a Union Oil Company proprietary sulfur removal process.

For evaluating the ASSP concept, a separate study was done to select the best amine process for concentrating gases from Unishale B and Lurgi. Although the conventional sulfur control processes proposed for Unishale B and Lurgi retorting are the Unisulf and DEA plus Stretford respectively, Methyldiethanolamine (MDEA) was selected to evaluate the ASSP concept because:

- It is partially selective for H_2S over CO_2. About 50% of the CO_2 will pass through, thus reducing solvent circulation requirements which in turn reduce both capital and operating costs.
- MDEA is not significantly degraded by COS.
- Hydrocarbon solubility is low.
- The process is non-proprietary.
- MDEA solvent is relatively inexpensive.
- No exotic metallurgy is required.

Design bases for each retort type are summarized in Table I.

COST ESTIMATES

Equipment costs for Lurgi, Unishale, and MIS commercial plants were taken from EPA Pollution Control Technical Manuals (PCTMs) which describe the Union [4]. Cathedral Bluffs [1] (Tract C-b), and Rio Blanco [5] (Tract C-a) projects in detail. All major ASSP equipment items were sized and costs were factored from in-house data bases as well as information contained in the PCTMs. All cost data were factored to first quarter 1985 using the CE Plant Cost Index. Equipment additions and deletions are summarized in Table 2.

Changes in capital and operating costs have been estimated as shown in Table 3.

DISCUSSION

Results of this study show that the best potential for application of ASSP are those processes which already have a spent shale combustor integrated into the retorting process (e.g., Lurgi, Unishale C, Chevron STB, and Tosco

TABLE 1. CONCEPTUAL DESIGN BASES

	Direct Heated		Indirect Heated	Integral Combustor	
Retorting Process	MIS	Unishale C	Unishale B	Lurgi	Unishale C
Site	--Tract	C-b--	Union	Tract C-a	Union
Plant Capacity, kg/sec	380	143	286	1251	286
, (TPSD)	(36,200)	(13,600)	(27,200)	(119,000)	(27,200)
Shale Grade, L/Tonne	112	146	142	96	142
, (GPT)	(27)	(35)	(34)	(23)	(34)
No. of Retorts	19	1	2	13	2
Conventional Sulfur Control:	Case A: FGD	Unisulf	Unisulf	DEA+	Unisulf
	Case B: Stretford	Unisulf		Stretford	
ASSP Parameters:					
Combustor Type	- -Fluid Bed- -		Fluid Bed	Lift Pipe	Fluid Bed
Number of Combustors	5		2	13	2
Temperature, K	1005		1005	922-977	1005
, (°F)	(1350)		(1350)	(1200-1300)	(1350)
Spent Shale Combusted, kg/sec	110		222	489	222
, (10^3 lb/hr)	(871)		(1760)	(8533)	(1760)
Raw Shale Combusted, kg/sec	8.5		16.8	0	16.8
, (10^3 lb/hr)	(67)		(133)	(0)	(133)
Retort Gases to Combustor, kg/sec	227.4		0	13.5	0
, (10^3 lb/hr)	(1805)		(0)	(107)	(0)
Acid Gases to Combustor, kg/sec	1.5		1.4	3.7	1.4
, (10^3 lb/hr)	(12)		(11)	(29)	(11)
Total Sulfur Fed to Comb., kg/sec	0.68		0.28	0.15	0.28
, (lb/hr)	(5373)		(2204)	(1160)	(2204)
Ca/S Molar Ratio	23		133	1088	133
Flue Gas SO_2, ppmv	*		*	30	*

*To be determined in Phase II Pilot Plant Program

TABLE 2. EQUIPMENT CHANGES FOR ASSP

	Direct Heated		Indirect Heated	Integral Combustor	
	Case A: FGD	Case B: Stretford			
Retort Process	Mis/Unishale C	MIS/Unishale C	Unishale B	Lurgi	Unishale C
Systems Added for ASSP:	----------MDEA----------		MDEA	MDEA	MDEA
	----------Fluidized Beds----------		Fluidized Beds		
Systems Deleted for ASSP:	Unisulf	Unisulf	Unisulf	DEA	Unisulf
	MIS FGD System	MIS Stretford	Recycle Gas Heater	Stretford	
	MIS Steam Boiler	MIS Steam Boiler	Spent Shale Cooler		

TABLE 3. COST COMPARISON FOR ASSP

	Direct Heated		Indirect Heated	Integral Combustor	
	Case A	Case B			
Retorting Process (Base)	MIS/Unishale C		Unishale B	Lurgi	Unishale C
Incremental Cap. Cost, 10^6 (Relative to Base)	-71.2	-63.2	+90.2	-13.0	-32.1
Incremental Annual Operating Cost, 10^6/yr*	+10.83	+12.07	-19.21	- 2.29	- 1.56

* 150 psig steam $8.81/tonne ($4.00/10^3 lb)
400 psig steam $9.91/tonne ($4.50/10^3 lb)
600 psig steam $11.01/tonne ($5.00/10^3 lb)
Fuel Gas $2.84/GJ ($3.00/10^6 Btu)
Electricity $0.014/MJ ($0.05/kWh)

HSP). Capital and operating cost savings for Unishale C and Lurgi cases are primarily a result of deleting the Unisulf and Stretford plants.

For indirect heated processes (e.g., Paraho Indirect, Tosco II, and Unishale B), payout on the additional investment for ASSP equipment would be good to marginal, depending primarily on how effectively the add-on spent shale combustor can be integrated into the process. That is, economically, it is better that heat from the combustor products be recovered and used directly in the retort rather than used for steam raising. For this study, it was assumed that combustor flue gases are used to heat recycle gas for the Union B retort (this has been proposed by Union as the preferred Unishale C scheme). The economics of this case are also quite sensitive to values used for steam and fuel. The values used here are considered quite conservative. Use of higher values will, of course, tilt the economics more favorably toward ASSP.

In the direct heated cases, results show substantial savings in capital costs for ASSP relative to both Stretford and flue gas desulfurization approaches. However, the capital savings are at the expense of increased operating costs primarily as a result of deleting the MIS steam boiler which produces much larger quantities of useable steam than can be produced by ASSP. Again, the economics here are quite sensitive to steam values although, in this case, higher steam values would tilt the economics away from ASSP. Also, investment in fluid bed equipment is very large (on the order of $130 million) because of the very large volumes of dilute MIS retort gases which must be handled. However, if fast fluid beds can be utilized, the number of additional combustors required could be reduced from four to as few as one or two. This would reduce capital investment significantly and tilt the economics more in favor of ASSP. There may also be an environmental advantage to using ASSP for Case B. The ASSP process should remove the non-H_2S sulfur species from the MIS retort off gas, while the alternative Stretford process would not effectively remove the non-H_2S sulfur species.

EXPERIMENTAL WORK TO BE PERFORMED

Phase II of the EPA contract will test the effectiveness of the ASSP concept in an integrated oil shale pilot plant utilizing a fluidized bed combustor. The 0.063 kg/sec (6 ton/day) plant incorporates the Tosco HSP technology and uses a 0.46 m (18 in.) diameter combustor which is an integral part of the retorting process. The test program is scheduled for October 1985, and a final report is anticipated early in 1986. Questions to be addressed in the Phase II test program include:

- How effective is ASSP in controlling sulfur emissions?
- Will ASSP produce large quantities of NO_x?
- What are the most favorable operating conditions to achieve maximum sulfur control while holding NO_x emissions to a minimum?
- Will retorted or raw oil shale combustion produce significant emissions of trace elements such as mercury or cadmium?

CONCLUSIONS

From the results of the Phase I study, it appears that the ASSP concept is technically and economically viable compared to conventional technologies for most retorting processes. However, some technical uncertainties will remain until further testing is conducted. Environmental acceptability depends not only on the effectiveness of ASSP for controlling sulfur emissions but also on holding NO_x emissions to a low level and on not volatizing significant quantities of trace elements such as mercury and cadmium.

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Kenneth Van Zanten is currently a project manager of J & A Associates, Inc., an independent laboratory doing R & D and analytical work for industrial and government clients. Prior to joining J & A, he held positions in process engineering and licensing, marketing, project management and environmental engineering with Supurtek, Ltd., Tosco Corporation, Marathon Oil Company and Dow Chemical Company. He received his B.S. in chemical engineering from the University of Colorado in 1964, and a M.S. in chemical engineering from the University of Denver in 1970.

Gerald R. Chiaramonte is currently a vice president of J & A Associates, Inc., an independent laboratory doing R & D and analytical work for industrial and government clients. Prior to joining J & A, he held positions in process engineering, project management, and engineering management with Tosco Corporation and Atlantic Richfield Company. He is a 1969 graduate of the University of New Mexico with a B.S. in chemical engineering.

Edward R. Bates, a Senior Physical Scientist for the USEPA, earned his B.S. and M.S. degrees in Geology from Michigan State University. Since joining the USEPA in 1977, he has specialized in environmental research related to oil shale development and is presently located with the Hazardous Waste Engineering Research Laboratory in Cincinnati, Ohio. He is currently serving as the manager for the oil shale program of the Air and Energy Engineering Research Laboratory, Research Triangle Park, North Carolina.

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